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Nanoporous Silicon Ignition of JA2 Propellant

by Stephen L. Howard, Wayne A. Churaman, and Luke J. Currano

ARL-TR-6950 **June 2014**

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14. ABSTRACT
Nanoporous silicon with sodium perchlorate oxidizer was examined as an igniter of bulk JA2 propellant. Electron microscopic postmortem examination of the propellant after the first experiment suggested the use of an additional igniter material. Nano bismuth oxide and nano cuprous oxide thermites were both attempted as supplemental igniter materials. The nano bismuth oxide thermite did not ignite the JA2, but the nano cuprous thermite did successfully provide ignition.

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nanoporous silicon, JA2 propellant, thermite, ignition

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1. Introduction

Nanoenergetic materials have been researched for years. A number of these materials take the form of the classical thermite—namely, an electroactive metal and a metal oxide. The oxygen exchange reaction typically liberates intense heat, light, and hot particles. The nanoparticle size has a profound effect on reactivity and speed of reaction (*1–11*). While silicon (Si) has been tried as an electron source for thermite-like reactions (*12*), it is when silicon particle size is in the nanometer-size range that the reaction rate becomes interesting.

Since many years of experience fabricating silicon into wafers exists in the electronics industry, the possibility of fabricating a wafer containing nanoparticles of silicon is an exciting concept. For example, Currano and Churaman in 2009 demonstrated on-chip nanoporous silicon preparation by electrochemical etch (*13*). In this preparation, a silicon wafer is electromachined to form a region of pores in the wafer that has wall thicknesses on the order of nanometers. These pores are filled with a strong oxygen donor (sodium perchlorate [$NaClO_4$]), and the reaction is initiated thermally. It is postulated that the large energy of reaction and the speed of the reaction would make a good igniter for propellant.

Other benefits could arise from having a propellant igniter fabricated as an integral element of a silicon chip. Integrated circuits that filter the firing command signal could remove extraneous electromagnetic signals that would satisfy the hazard of electromagnetic radiation to ordnance (HERO) requirements of modern munitions. Such integrated circuits can be made in large numbers rather inexpensively and, as such, could be located at multiple locations in a munition. The firing of each circuit could be timed appropriately to effectuate a distributed ignition that optimizes the stored energy transfer of the propelling charge to a projectile. This study is one of the first steps to realize this type of integrated circuit igniter—namely, to investigate the ignition of propellant by the on-chip igniter.

2. Experimental

The nanoporous silicon chips for this study were prepared by an electrochemical etch (*13–15*). This method provided an etched region on the chip with a multitude of fine pores in the silicon. An electrical bridgewire was deposited on the chip in the vicinity of the etched nanoporous region. The chips were then mounted on a dual in-line package (DIP) socket so that external electrical leads could easily be attached (figure 1).

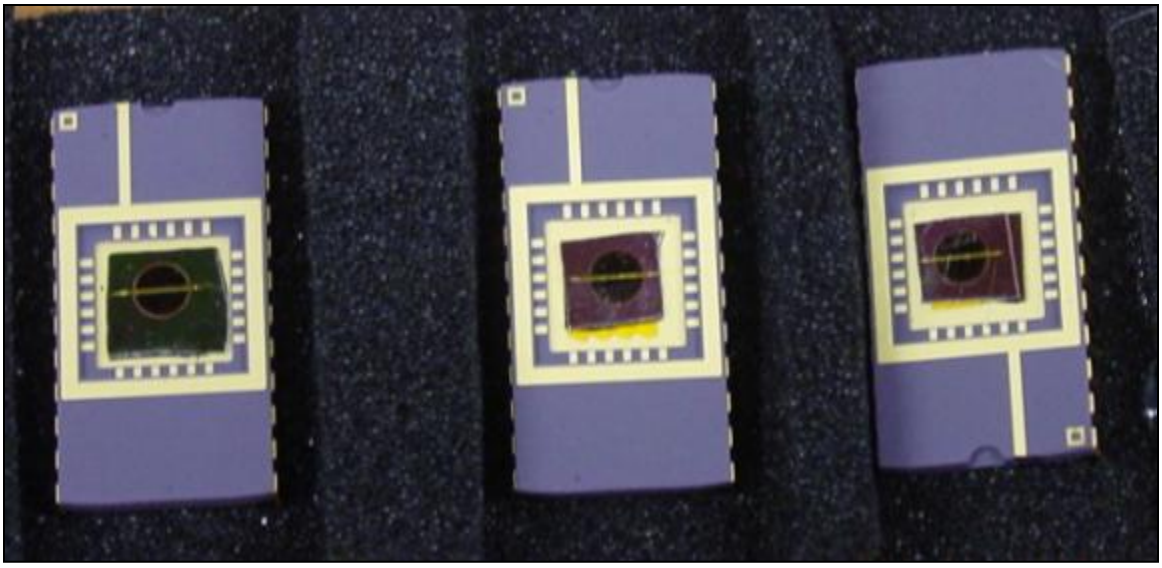

Figure 1. Photograph of three activated nanoporous silicon chips loaded with $NaClO_4$ mounted on DIP sockets resting on an electrically conductive foam pad prior to experiments.

In order to "activate" the chip, several drops of a liquid oxidizer solution of $NaClO_4$ dissolved in methanol were placed on the etched region of the chip and allowed to penetrate and wet the pores before the methanol evaporated. This activation deposited solid oxidizer ($NaClO_4$) inside the porous silicon, so everything was contained on/in the chip. Since $NaClO_4$ is hydroscopic, application of the solution and the attendant drying were performed in a nitrogen-purged glovebox for low humidity.

Electrical leads were then attached to the activated chips, and each chip was placed singly in a transparent, flexible polyvinyl chloride (PVC) container. The PVC container (see figure 2) contained the dry nitrogen atmosphere from the glovebox during hand-carry transportation from the preparation room to the explosives-rated experimental chamber room. The electrical leads on the outside of the PVC container were connected to the firing circuitry (an impressed voltage of 3 V across the chip bridgewire was sufficient to activate the silicon/perchlorate reaction), and the transparent portion of the chamber allowed us to view the ignition event by high-speed camera. High-speed video was obtained at 21,000 frames per second with a Phantom 5 high-speed camera.

Both transparent and regular 13-mm outer diameter by 3-mm-thick JA2 disks (see figure 3 for visual view and figure 4 for scanning electron microscope [SEM] images of the surfaces of both disks, showing that transparent and regular forms of JA2 are not discernible under the electron microscope) were prepared as ignition source receptors. The bismuth trioxide (Bi_2O_3) and copper (II) oxide (CuO) thermite powders were prepared by standard laboratory procedures from the respective metallic oxide and nano aluminum powder (*16*). Electron micrographs were obtained with an International Scientific Instruments, Inc., SS40 SEM. The electron energy was 20 kV.

Figure 2. Photograph of the activated nanoporous silicon chip in the PVC container showing attached firing leads; JA2 propellant disk rests over activated region of chip under electrostatic protective aluminum foil.

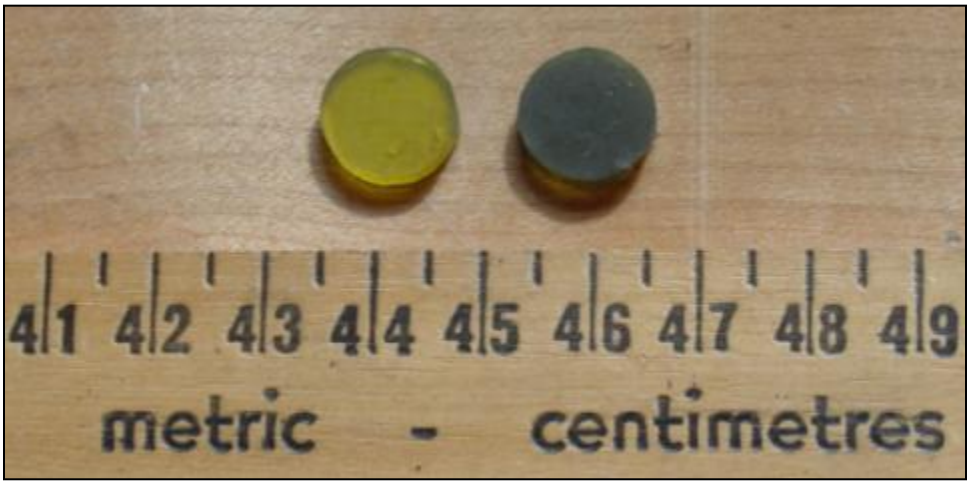

Figure 3. Photograph of transparent and regular JA2 disks for ignition experiments.

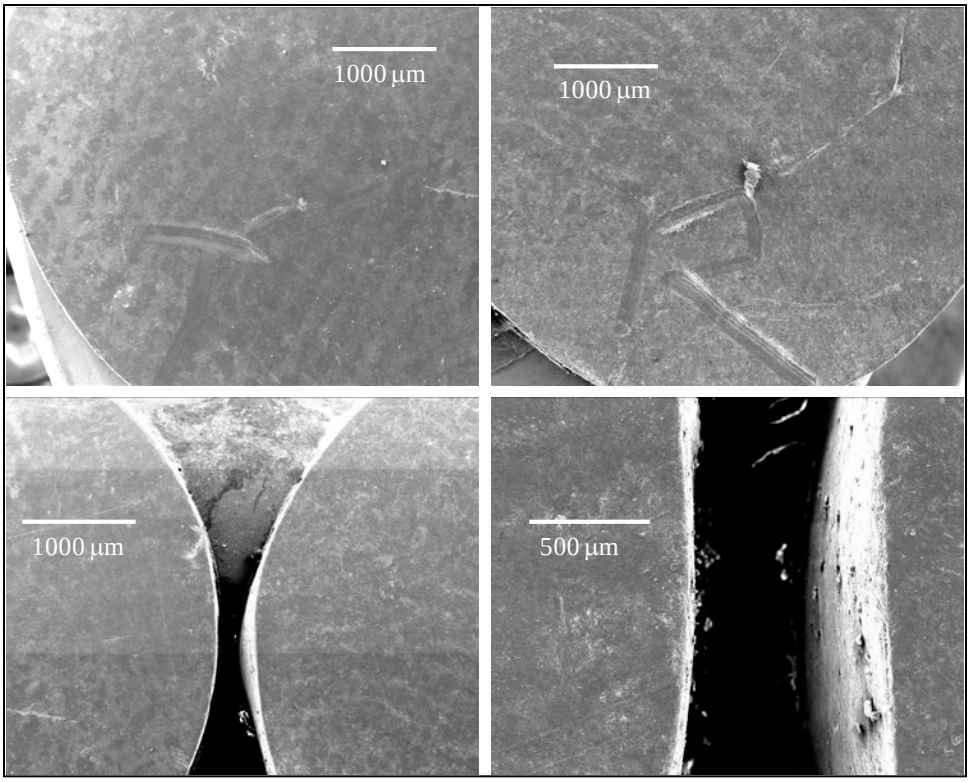

Figure 4. SEM images (at 10× magnification) of transparent (marked with "T") and regular (marked with "R") JA2 disks for ignition experiments showing few discernible features; lower image shows both disks (transparent [T] on left).

3. Results and Discussion

Previous open-air initiations of the on-chip silicon/perchlorate reaction resulted in large plumes of bright material rapidly emanating from the chip (*13*, *15*). It was expected that such a hot plume of gases and particles would easily ignite JA2 propellant. Since it was not known a priori if the major energy transfer causing ignition would be hot particles and hot gases or the intense radiation from the reaction, two forms of JA2 propellant were provided (see figure 3). Regular JA2 has several tenths of a percent of dark carbon particles in the formulation. This form of JA2 would be nearly opaque to radiation (an unknown spectral distribution of ultraviolet to near-infrared) emanating from the reaction site. The radiation would heat the JA2 and accelerate the ignition as demonstrated in plasma electrothermal-chemical ignition (*17*). Transparent JA2, also provided for the experiments, did not contain radiation-absorbing carbon particles and might have a weaker response to ignition if the principal method of energy transfer was by radiation. If the principal energy transfer mechanism involved the hot particles and hot gases, the ignition of the two forms of JA2 would be comparable.

Figures 5 and 6 show frames from the high-speed video with a regular JA2 disk or a transparent JA2 disk, respectively, placed immediately over the etched region of a chip and held in place with alligator clips holding the electrostatic shield (see figure 2). The view is through the transparent PVC container to maintain the low-humidity environment. Since the DIP sockets were inside the PVC container and not available after leaving the glovebox for manual manipulation for the view of the camera, not all the experiments show the DIP socket on the horizontal. The experiments were not affected by the attitude of the socket while in the PVC container. An overlay was placed over a frame most clearly locating the elements of the chip/propellant assembly. Red rectangles locate the electrical alligator leads that are on opposite sides of the active chip region, the green line locates the edge of DIP, the blue square locates the chip, and the yellow oval locates the JA2 disk. Light emission from exiting reaction products was only visible after exiting from the edge of the propellant disk or the aluminum shield.

Both JA2 experiments showed intense light when the $Si/NaClO_4$ reaction was initiated. However, the pressure above the reaction pushed the JA2 disk away without igniting. The experiments were repeated with alligator clips holding the disks in place. Figure 7 shows the chip surface and the JA2 disk surfaces after recovery from the PVC container. For both JA2 disks, surface damage appeared to be minimal. Views of the surface under the electron microscope in figures 8–10 show particles of Si of various sizes and shapes but little damage to the JA2 surface itself (compare to figure 4, micrographs of the surfaces of untested initial JA2 disks). Therefore, we concluded that hot gas and/or particles would be the dominant mechanism for ignition if the JA2 were sufficiently attached to the chip so that it could not move.

Since the $Si/NaClO_4$ reaction appeared to be intense, but short in duration and low in total integrated energy transfer, a secondary igniter material placed between the chip and the propellant could overcome these difficulties. Both Bi_2O_3 and CuO nanothermite powders were available at the time of the chip experiments. Both nanothermites produce copious amounts of hot metal droplets as well as hot gas. The bismuth thermite has been hailed as an excellent ignition material by several sources (*18–21*). Therefore, both nanothermites were investigated as a secondary igniter material.

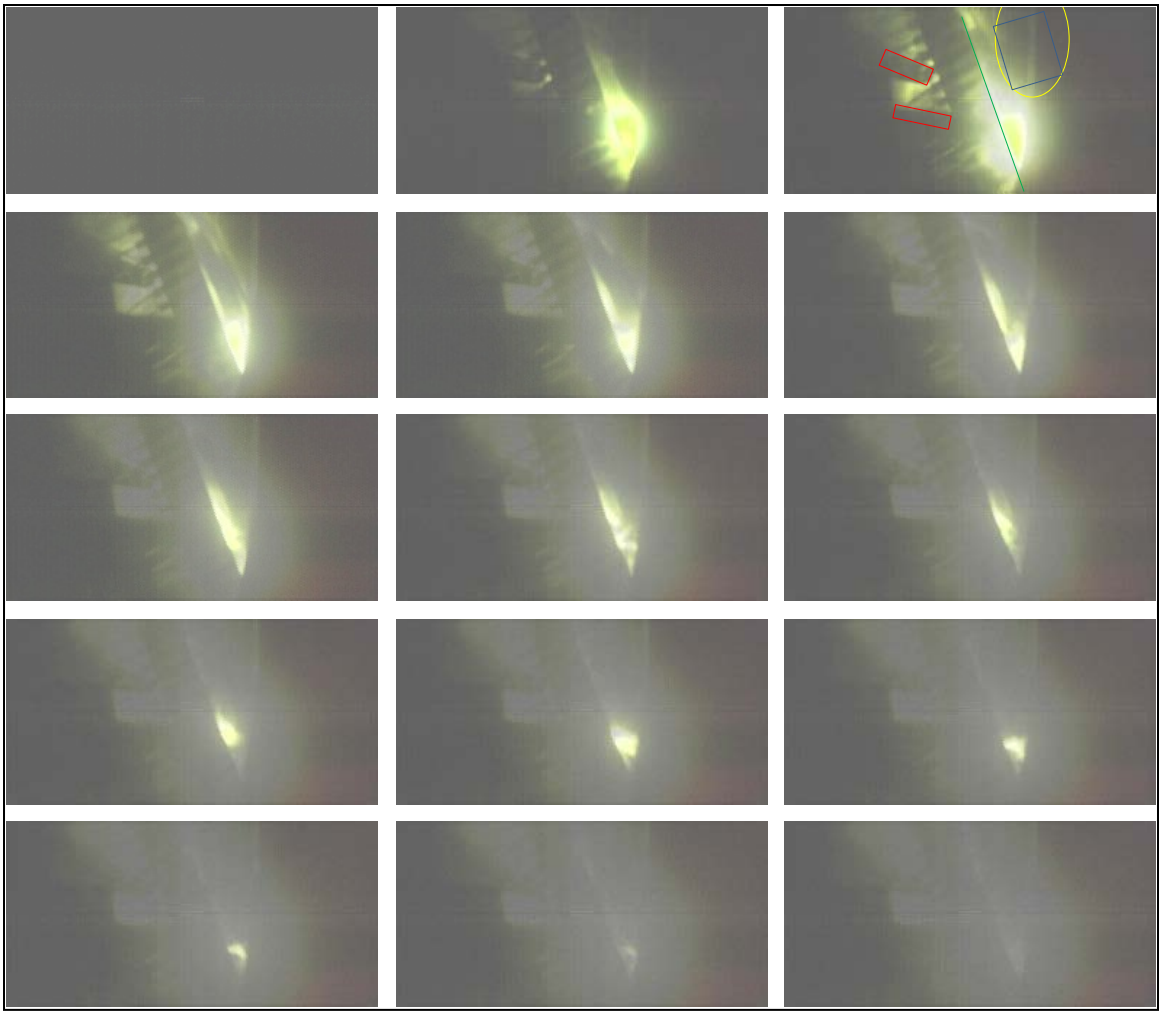

Figure 5. High-speed video frames (from left to right and top to bottom at 47 μs/frame) showing on-chip reaction of Si/$NaClO_4$ against regular JA2 (red rectangles locate the electrical alligator leads, green line locates the edge of DIP, blue square locates the chip, and yellow oval locates the JA2 disk); light emission is from reaction products leaving reaction site.

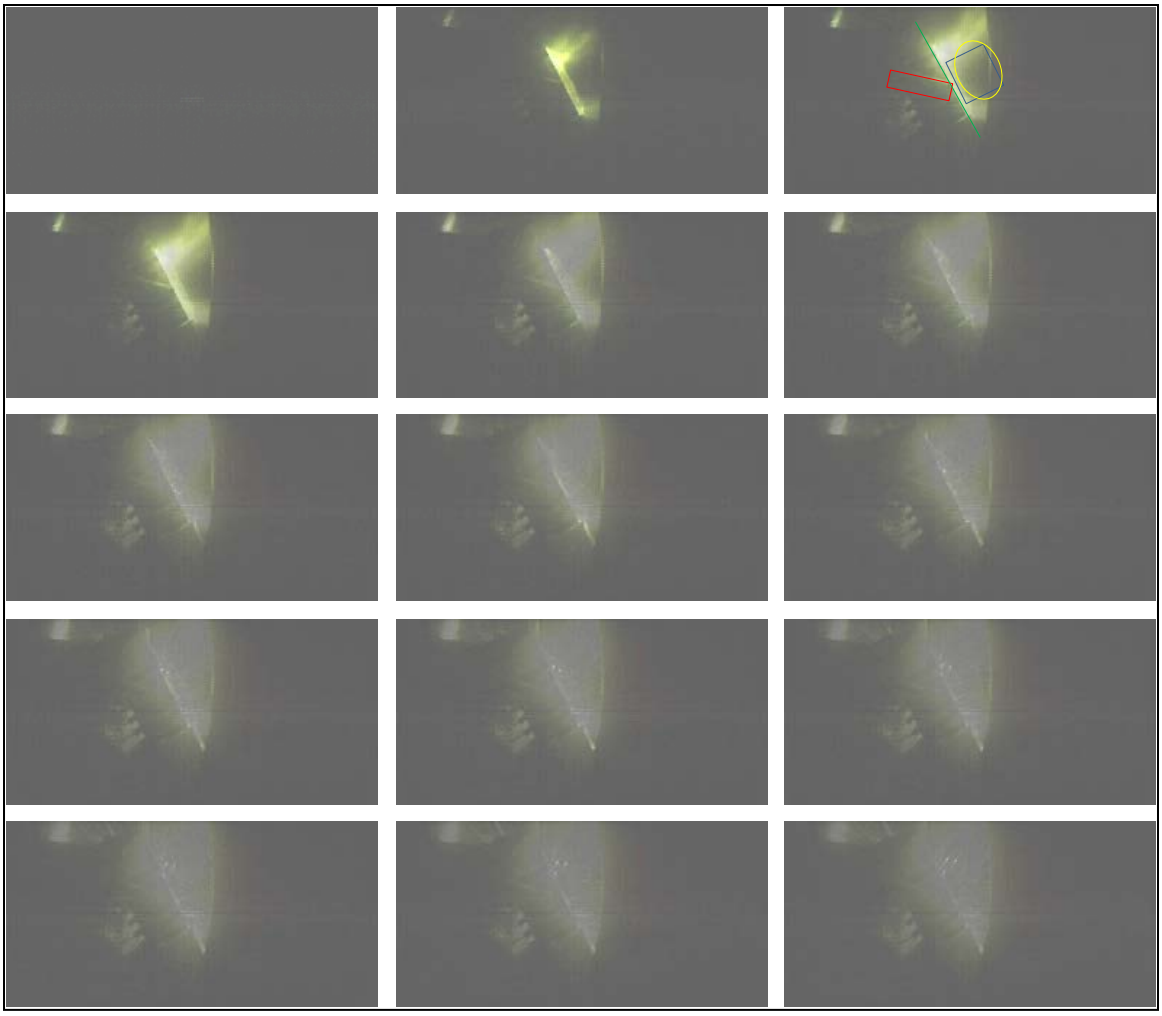

Figure 6. High-speed video frames (from left to right and top to bottom at 47 μs/frame) showing on-chip reaction of Si/$NaClO_4$ against transparent JA2 (red rectangle locates the electrical alligator lead, green line locates the edge of DIP, blue square locates the chip, and yellow oval locates the JA2 disk); light emission is from reaction products leaving reaction site.

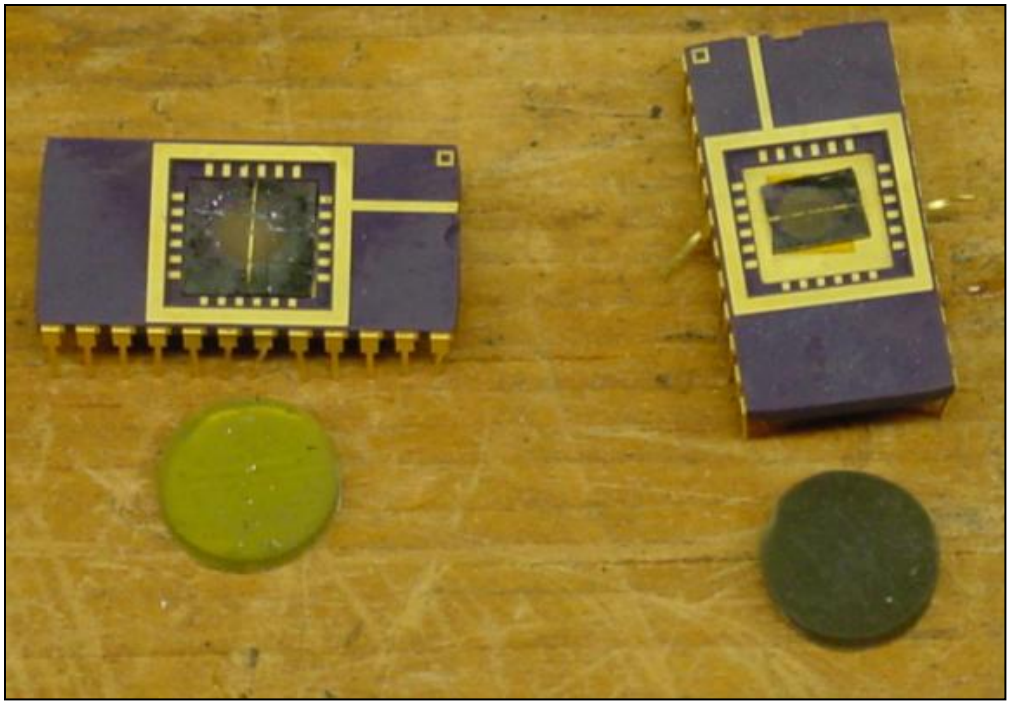

Figure 7. Photograph of regular and transparent JA2 disks after initiation of the $Si/NaClO_4$ reaction showing Si particles on the disk with minimal damage to the disk; chip surface also shown with reaction zone visible.

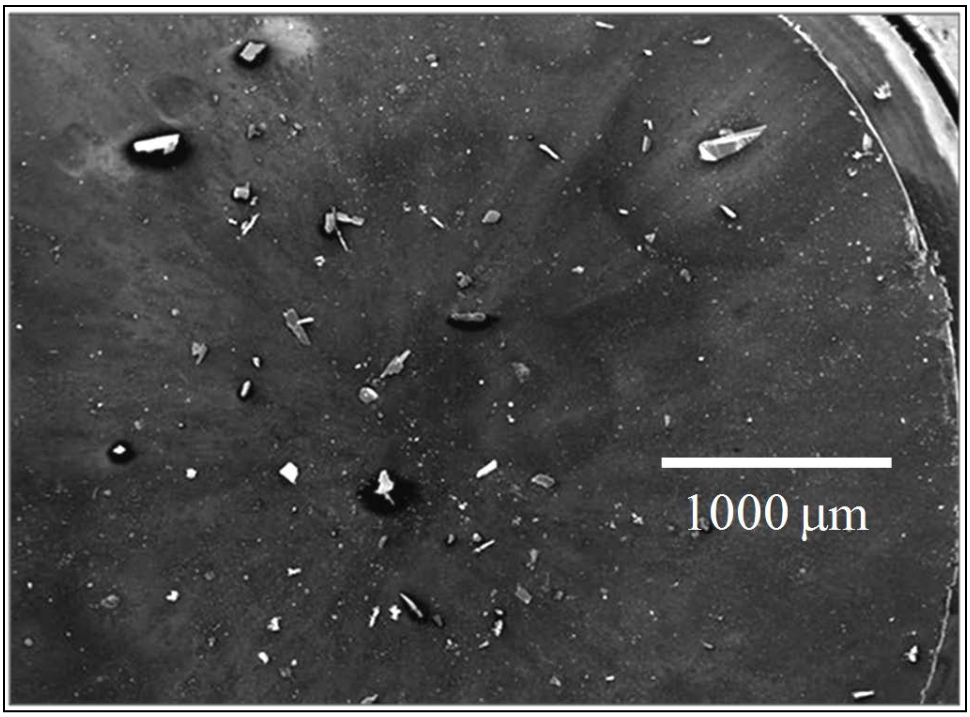

Figure 8. JA2 surface at 13× magnification showing Si particles and splash rays from the expanding reaction products of the $Si/NaClO_4$ reaction.

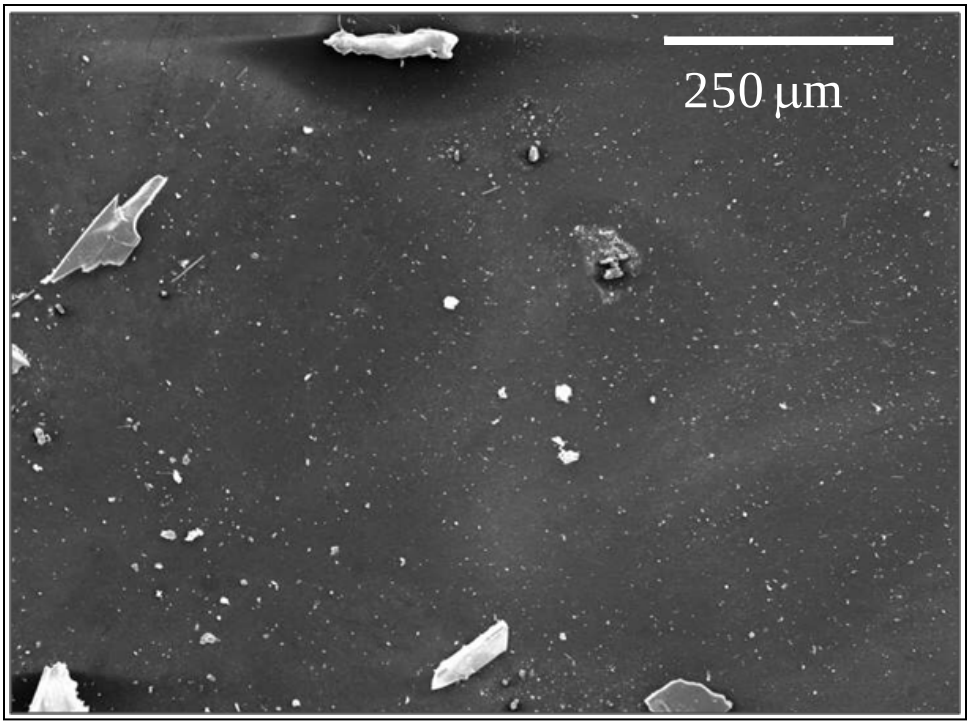

Figure 9. JA2 surface at 40× magnification showing Si particles in a splash ray region showing minimal damage on surface.

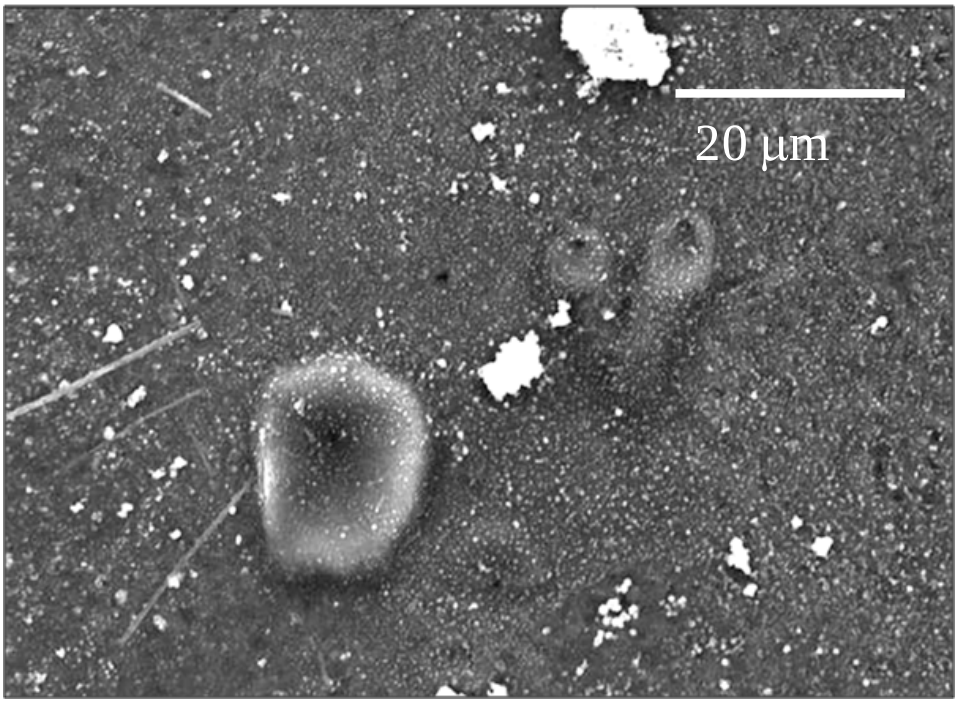

Figure 10. JA2 surface at 500× magnification showing Si particles and several thermal "blooms" where JA2 combustion reactions started but were insufficient to sustain.

The Bi_2O_3 nanothermite was investigated first. It has been touted as a good ignition material, safe, environmentally friendly, etc. (*18–21*). Since other ignition studies had been performed successfully in this laboratory (*16*) with approximately 0.1 gm of nanothermite mixture, this amount of Bi_2O_3 nanothermite was placed above the active region on the chip between it and the JA2 disk (regular JA2 was used for all the nanothermite experiments).

As shown in figure 11, the output from the nanothermite enhanced the igniter output. However, as shown in figure 12, the JA2 did not ignite. This result was not expected because of previous experience with this thermite and JA2 propellant. Figures 13 and 14 show electron microscope images of the surface of the JA2 disk showing some damage. Most of the damage on the propellant surface was from hot gas flow. The erosion and video images showed that the gas was moving at high velocity. It is possible that the high-velocity gases removed hot particles that would have otherwise interacted with the surface, creating hot-spot ignition sites. If the high-velocity gases removed the combustion intermediates and flame zones faster than the flame-zone chemical reactions could provide feedback into the surface for further reaction, the solid propellant would not have ignited successfully.

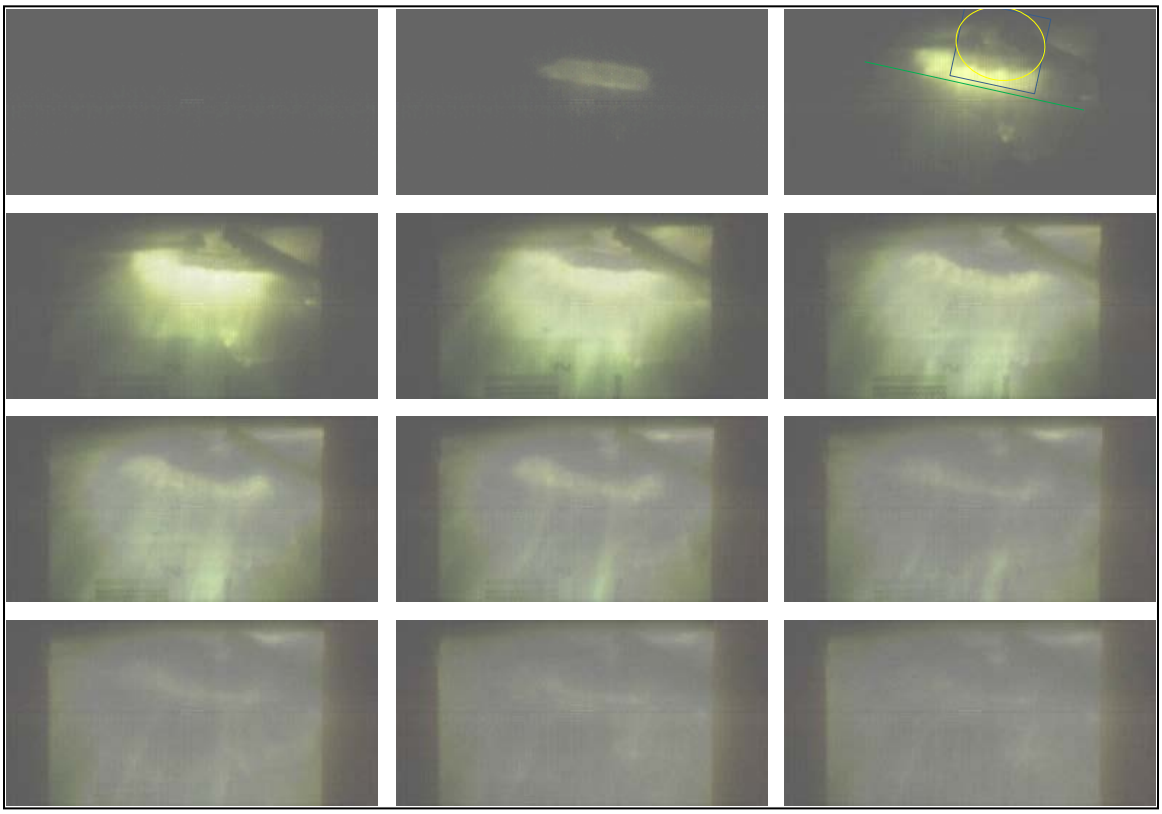

Figure 11. High-speed video frames (from left to right and top to bottom at 47 μs/frame) showing on-chip reaction of $Si/NaClO_4$ with Bi_2O_3 nanothermite powder against regular JA2 (green line locates the edge of DIP, blue square locates the chip, and yellow oval locates the JA2 disk); light emission is from reaction products leaving reaction site.

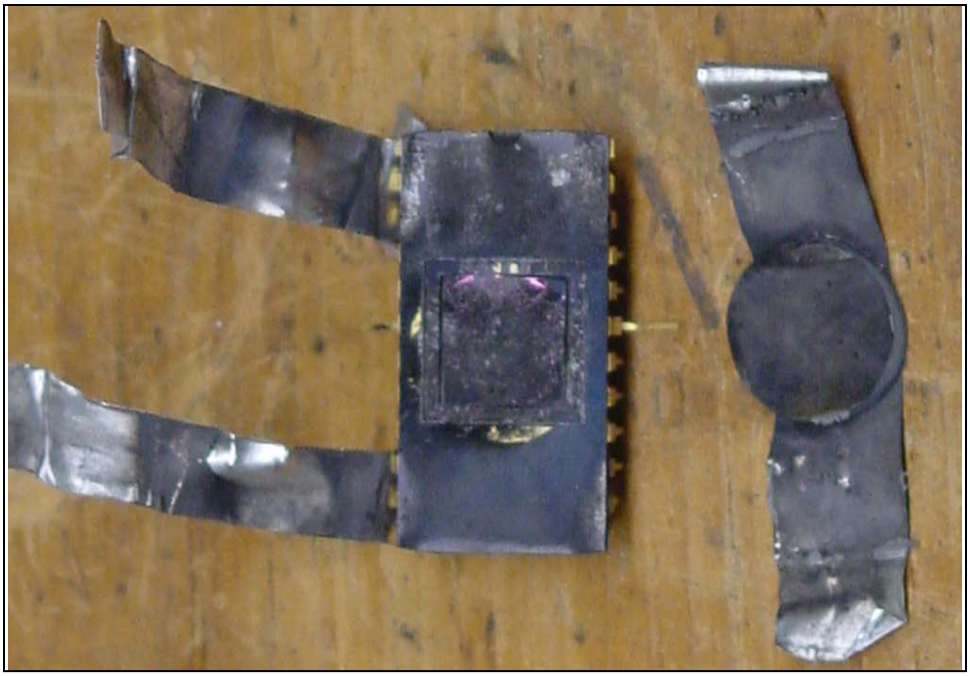

Figure 12. Photograph of Bi_2O_3 nanothermite reaction residue on chip and JA2 disk after ignition attempt.

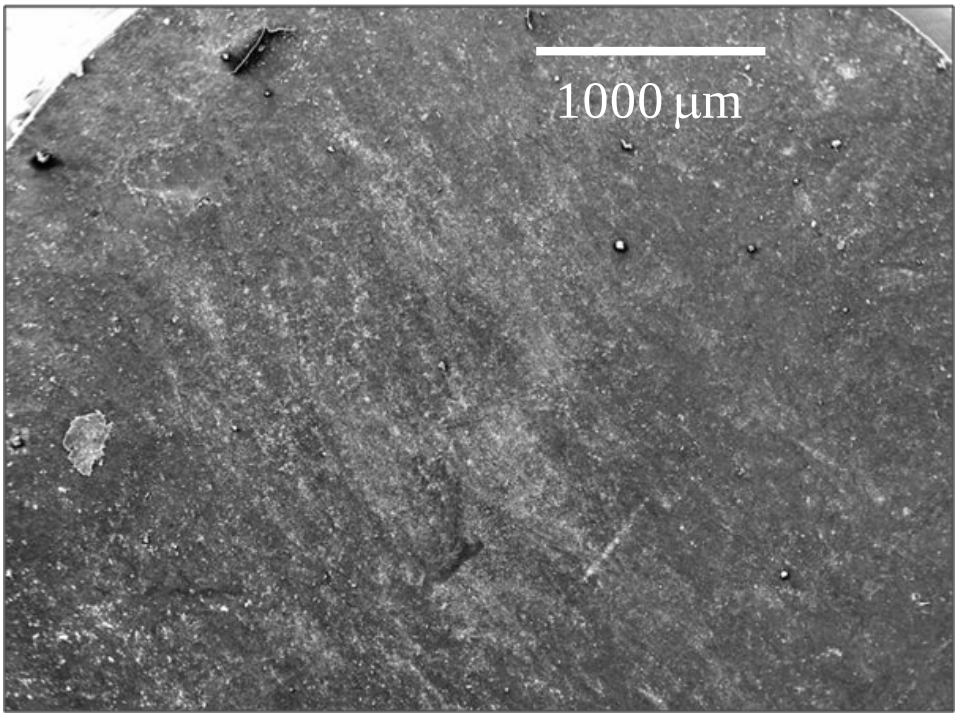

Figure 13. JA2 surface at 12× magnification showing Bi droplet particles and erosion areas from gases moving from reaction site.

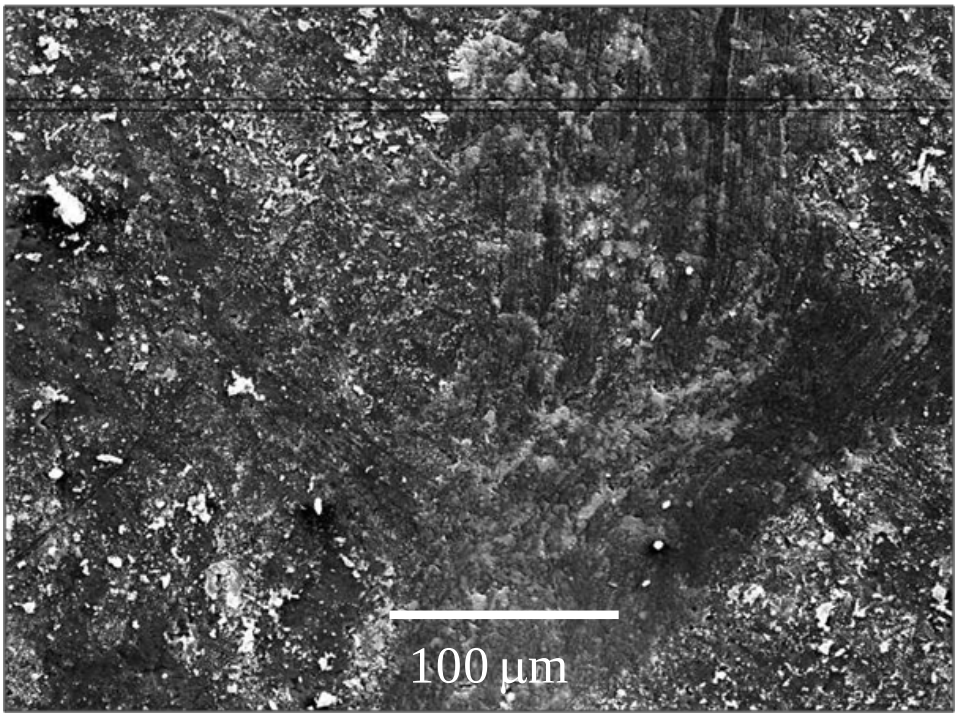

Figure 14. JA2 surface at 100× magnification showing Bi droplet particles and erosion areas from gases moving from reaction site.

CuO thermites (both nano and micron sized) also had been used in previous ignition studies at the U.S. Army Research Laboratory (*22*, *23*). The same mass of CuO nanothermite (0.1 gm) was placed on a chip in similar fashion as the Bi_2O_3 nanothermite. Frames of the high-speed video are presented in figure 15. The CuO nanothermite reaction was more intense and longer in duration than for the Bi_2O_3 nanothermite. After the initial frames, some of the luminosity was due to the combustion of the JA2 disk. The remains of the chip and aluminum foil shield are shown in figure 16. Only a slight amount of JA2 remained on the aluminum foil backing afterwards. This amount of leftover JA2 on the foil was not unexpected since the experiment was conducted at nearly atmospheric pressure when the burning rate of JA2 is quite low. At this pressure and with the aluminum foil backing of the disk as a heat sink to lower the temperature, quenching of the combustion with residual propellant would be typical.

Therefore, the on-chip $Si/NaClO_4$ reaction could be used as an ignition source for propellant. However, the initial reaction would need to be augmented by a small amount of an additional igniter material. This igniter material may need to be matched with the propellant type for optimal performance. Since the initial reaction is on a silicon chip, additional protective circuitry (i.e., HERO compliant) could be built on the chip for little extra cost.

As a generality, reduction in the footprint (volume of the material itself and the volume of the containment thereof) of igniter material also can reduce the sensitivity potential of a round (*24*). However, the insensitive munition (IM) compliancy of a munition is ultimately determined by many tests of an integral round, with some tests comprising groupings of multiple rounds.

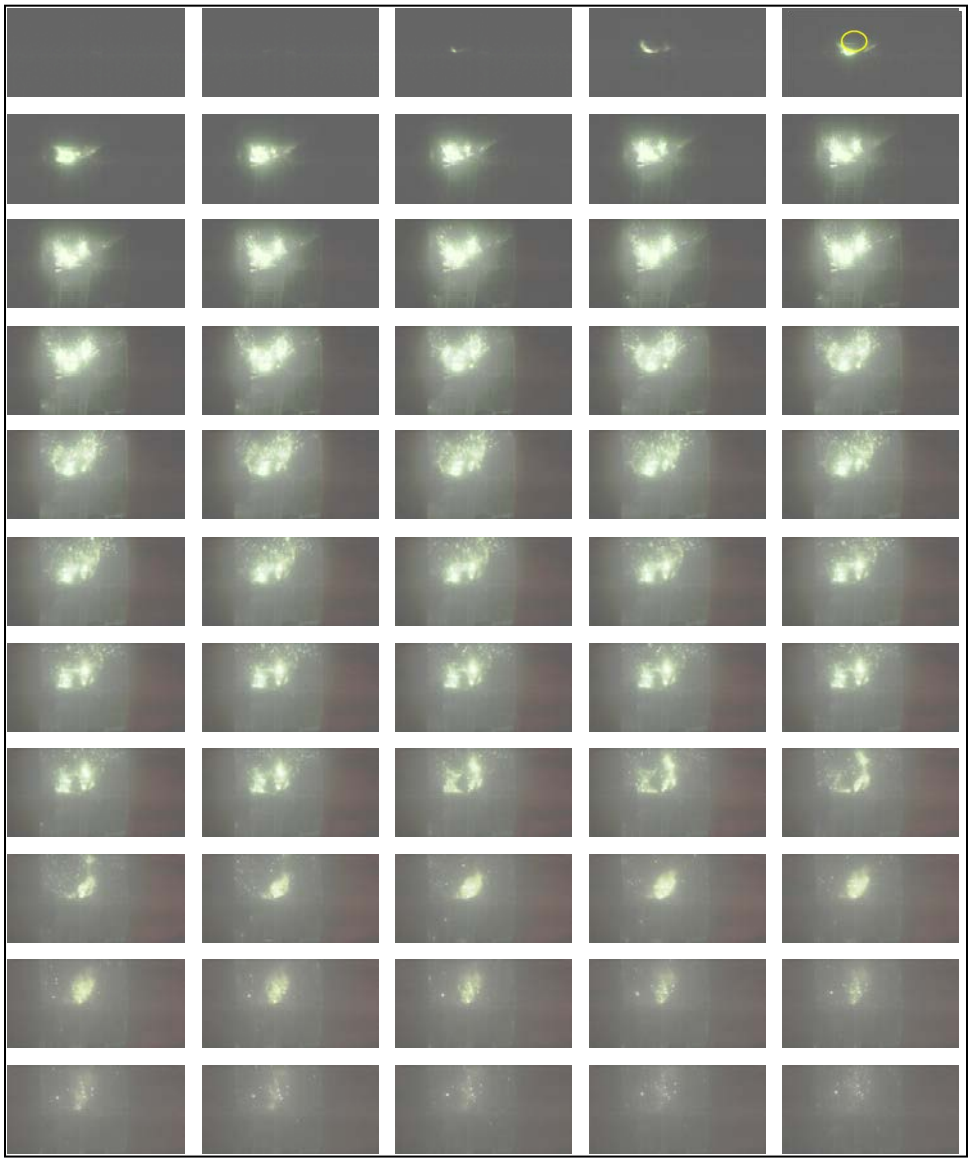

Figure 15. High-speed video frames (from left to right and top to bottom at 47 μs/frame) showing on-chip reaction of Si/$NaClO_4$ with CuO nanothermite powder against regular JA2 (yellow oval locates the JA2 disk); light emission is from reaction products leaving reaction site as well as combustion of JA2 disk.

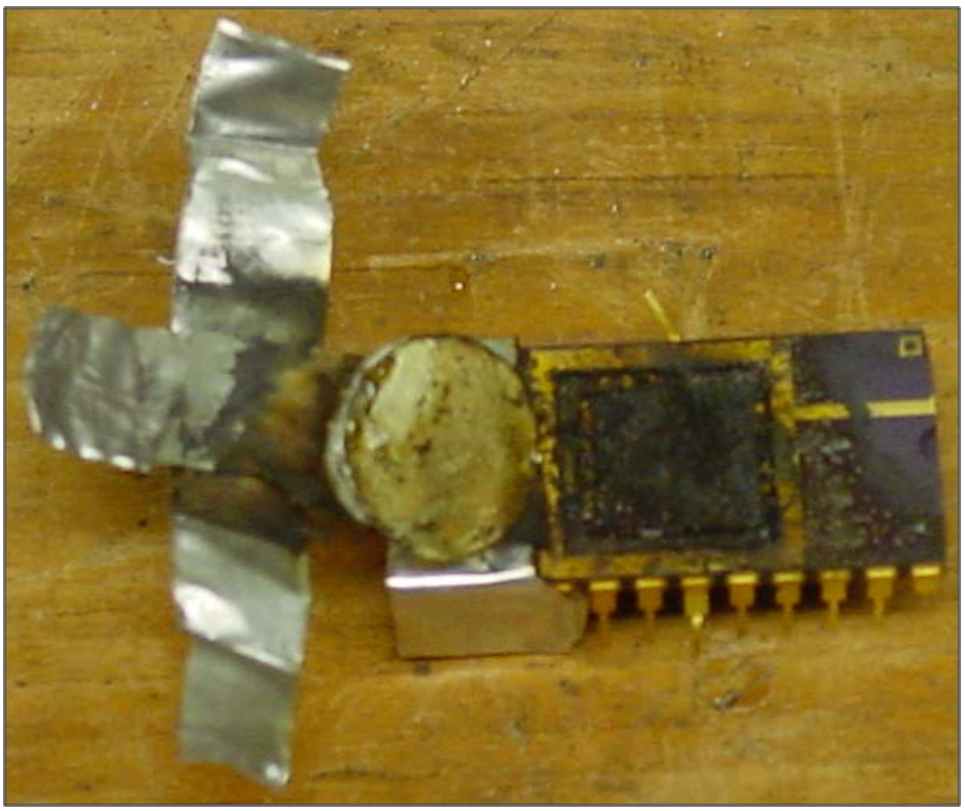

Figure 16. Photograph of CuO nanothermite reaction residue on chip and minimal JA2 disk residue after ignition.

4. Summary and Conclusions

The on-chip reaction of the fuel nanoporous silicon and the oxidizer $NaClO_4$ has shown promise as an ignition source. There is a large heat release with a short ignition delay. The fabrication is relatively easy and can be performed cheaply in large numbers using standard integrated-circuitry machinery common in the electronic industry. Since the nanoporous silicon can be made part of an integrated circuit during the course of fabrication, HERO protection can be built into the chip. More than one chip could be used in a particular round for programmed ignition to optimize the round's performance. These are all desirous attributes for high-performance, IM-compliant munitions.

In this study, this promising ignition source was examined for its ignition potential against JA2 propellant. JA2 was chosen because of its relative ease of ignition near atmospheric pressure. This propellant was also available in flat sheet form, which was the form factor most compatible with the flat surface of the silicon chip mounted on the DIP socket.

While the on-chip Si/$NaClO_4$ performed as previously demonstrated, the reaction was too rapid and the energy release too low for ignition of the propellant disk. An ignition enhancer of nanothermite was added between the Si/$NaClO_4$ reaction region of the chip and the propellant disk. Both Bi_2O_3 and CuO nanothermites were used. The Bi_2O_3 nanothermite failed to ignite the propellant. The CuO nanothermite did ignite the propellant. While it was anticipated that both nanothermites would ignite the propellant, the Bi_2O_3 nanothermite produced more gas product than the CuO nanothermite, which appeared to have removed hot particles and ignition chemical intermediates from the propellant surface more rapidly than required for successful ignition.

This study showed that the earlier touted benefits and possible advantages of on-chip ignition could be realized in a propelling charge munition. However, the on-chip device would not be an adequate ignition source by itself. An ignition train that contained a small amount of ignition booster that could be optimized for each propellant and/or application would be required.

We concluded that ease of fabrication of the on-chip igniter should provide a relatively inexpensive igniter. The on-chip circuitry could contain HERO protection without significant additional cost, weight, or volume. More than one chip could be used in a particular round for distributed ignition to optimize the round's performance. IM-compliancy of a particular munition may or may not be enhanced because of the presence of a smaller amount of igniter material.

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